INFLUENCE OF CURING-LIGHT BEAM PROFILE NON-UNIFORMITY ON DEGREE OF CONVERSION AND MICRO-FLEXURAL STRENGTH OF RESIN-MATRIX COMPOSITE

by

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DEDICATION

This thesis is dedicated to my parents and siblings, for their unlimited support, encouragement, inspiration and prayers.

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INTRODUCTION

An estimated 260 million resin-matrix composite (RMC) restorations are placed annually around the world.[1] A recent Cochrane Systematic Review revealed that the failure rate of posterior RMC restorations was twice that of posterior amalgam restorations.[2] Also, the median longevity of posterior RMC restorations was six years,[3-5] with bulk fracture and secondary caries the most common causes of failure.[2,4,6,7]

The degree of conversion (DC) has a significant impact on the performance of RMC restorations. It was found that DC is positively correlated with increasing the amount of radiant exposure received by RMC.[8] Conversely, unreacted monomers may plasticize the polymer structure, degrading the mechanical properties of RMC restorations.[9,10]

The irradiance can be defined as the radiant power per surface of known dimensions (mW/cm^2). However, the radiant exposure is the total amount of energy delivered to a resin-matrix composite surface during the entire irradiation procedure (J/cm^2).[11]

Previous studies have investigated the wavelength distribution and light intensity of light curing units (LCUs) and found that many light emitting diode (LED) LCUs do not deliver their intended light output.[12-14] In addition, the off-center position of the LED chips of some multiple emission peak LED LCUs may affect the beam profile uniformity.[15-17] Therefore, several laboratory studies have examined the light beam profile uniformity of different LCUs and revealed that the irradiance (mW/cm^2) and

spectral emission (nm) distribution were non-uniform across the LCU tip and may result in non-uniform polymerization of RMC restoration.[11,11,15-20] This may negatively affect the properties and longevity of the restoration.[16-18, 21-23] A clinical study reported a direct correlation between insufficient light exposure and decreased wear resistance of RMC.[24] Additionally, a direct relation was found between irradiance and the rate of free radical production.[25] Thus, appropriate irradiance and spectral emission delivered from the LCUs is required to obtain an optimum number of free radicals and achieve adequate polymerization of a RMC.[19,26-28]

Material properties, such as fracture resistance and elasticity, are often evaluated by flexural strength, flexural modulus and fracture toughness.[29] The flexural strength test is performed to predict the mechanical behavior of RMC because it resembles the compressive and tensile forces that are exerted in the stress concentration area and around the point of loading.[30] Additionally, it was reported that appropriate curing of RMC could improve the material's flexural properties.[31,32]

Several previous studies have evaluated μ-flexural strength of dental adhesive systems[33-35] using RMC specimens of smaller dimensions than suggested in the ISO 4049 flexural strength standard.[32,36,37]

The aim of this study was to evaluate the effect of the beam profile of a single and multiple emission peak LED LCU on the DC and μ-flexural strength at different light curing locations (LCLs) compared with quartz tungsten halogen (QTH) LCU.

HYPOTHESES

Null Hypothesis

Changing the horizontal location of LED LCUs will have no significant effect on the DC and micro-flexural strength of RMC compared with a QTH LCU.

Alternative Hypothesis

Changing the horizontal location of LED LCUs will have a significant effect on the DC and micro-flexural strength of RMC compared with a QTH LCU.

REVIEW OF LITERATURE

The development of LCUs allowed for the curing on demand of dental restorative materials.[26] Even though they have brought this advantage to dentistry, many issues associated with LCUs exist, such as confusion about adequacy of irradiance, radiant exposure, and exposure duration that may affect the polymerization of RMC.[26,38]

LIGHT CURING BEAM PROFILE NON-UNIFORMITY

Light beam non-uniformity has been significantly associated with the introduction of the LED LCUs and is caused by non-uniform distribution of spectral emission across the LCU tip.[20] Similar discrepancies were detected with ultraviolet (UV) and visible LCUs in1983 and 1985, respectively.[39,40] It was proposed that moving the LCU tip during photoinitiation might result in a more uniform polymerization of RMC restorations.[41] However, it was found that circular motion of the LCU tip resulted in non-uniform distribution of the hardness values across RMC specimens.[42]

Several methods are available to measure the light output of LCUs.[15,43] Of these, the light beam analyzer can accurately evaluate the power distribution across the LCU tip by taking a snapshot of the light source. Then, the average power value can be calculated at a specific area in the light beam. This value is termed the top hat factor, which represents the uniformity of the power distribution across the LCU tip.[44,45]

In 2010 Price et al.[46] evaluated the effect of using different light guides on the irradiance distribution and uniformity from different LCUs. They showed that the irradiance was non-uniform across the LCU tip and it differed significantly among the

LCU products and the types of light-generating methods. Furthermore, the light beam non-uniformity varied greatly when using different light guides on the same LED LCU body. Also, the authors concluded that a single irradiance value does not reflect the differences in irradiance across the LCU tip when characterizing a specific LCU. In addition, the irradiance distribution, the overall irradiance, and the top hat factor are significant factors that should be included when describing the irradiance of LCUs.[46]

Some LCUs with a broad spectral emission such as QTH and plasma arc (PAC) may not show significant differences in the wavelength distribution across the light beam.[16,20] On the other hand, due to the off center position of the chips in LED LCUs, the wavelength distribution can be non-uniform across the LCU tip.[24]

Price et al.[21] evaluated the irradiance uniformity across the LCU tips of multiple emission peak LED LCUs at two different spectral emission wavelengths. A significant difference was found in the irradiance uniformity between 405 nm and 460 nm wavelengths, which is mainly dependent on the location of the LED chips and their emission wavelengths within the LCU. Also, the authors suggested that the spectral radiant power should be included to characterize the light output along with the top hat factor and the irradiance distribution.[21]

In 2014 Michaud et al.[16] also evaluated the localized irradiance and wavelength distributions of four LCUs, one PAC, one single emission peak, and two multiple emission peak LED LCUs. The irradiance was found to be non-uniform with all LCUs. However, a PAC LCU had the most uniform irradiance distribution across the LCU tip, while the most non-uniform irradiance and spectral emission distribution occurred with multiple emission peak LED LCUs. The authors also concluded that the irradiance and

spectral emission of LCUs should not be reported as a single value as it will not precisely describe the output of the LCUs. Instead, they recommended providing an image to illustrate the irradiance and spectral emission distribution across the LCU tip.[16]

Price et al.[19] also investigated the effect of distance on the irradiance and light beam uniformity of four LED LCUs. In this study, the irradiance at the center of the light beam was measured at 1 mm and 9 mm distances between the LCU tip and a 3.9-mm diameter irradiance probe. The light beam uniformity was determined by calculating the mean top hat factor (measured at 2-, 4-, 6-, and 8-mm distances from the LCU tip). The light beam uniformity, the irradiance at the center of the light beam, and the top hat factor varied significantly among all LCUs. Therefore, the authors concluded that 1) Increased distance between the LCU and RMC might result in suboptimal polymerization, specifically with deep restorations, and (2) The light output cannot be represented by a single irradiance value.[19]

In 2014 Megremis et al.[20] investigated seven LED LCUs and included a QTH LCU as a reference point for all the tests. In this study, the irradiance, spectral distribution and the light beam uniformity were evaluated. The irradiance distribution of all LED LCUs was non-uniform across the LCU tip relative to the QTH LCU and it was recommended that clinicians should consider the location of the high irradiance regions on the LCU tip relative to the size of RMC restoration. Also, the position of the LCUs might significantly influence the radiant power and the wavelength received by RMC restorations.

A few studies have evaluated the effect of the light curing beam profile non-uniformity on DC and RMC physical properties.[15,17,18,23,44] However, no studies (*in vivo*

or *in vitro)* were found to evaluate the effect of the light beam non-uniformity on the mechanical properties of RMC restorations. In 2005 Vandewalle et al.[44] evaluated the influence of the light dispersion of QTH and LED LCUs on the DC of RMC. In this study, the DC was significantly influenced by the divergence angle of the light beam emitted from LCUs, as the lower the light dispersion, the higher the polymerization of RMC at greater distances. However, the DC values with LED LCUs were found to be either similar or better compared to QTH LCU at 5-mm distance between RMC and the LCU tip. Similarly, Arikawa et al.[15] measured the effect of light beam profile non-uniformity on RMC surface hardness. In this study, three QTH, one PAC, and one LED LCU were investigated. The light intensity varied significantly among all LCUs at different locations across the LCU tip. However, for all LCUs, the distribution patterns of the surface hardness corresponded roughly with light intensity. Also, compared to LED, QTH and PAC showed significantly greater non-uniformity in light intensity distribution. Furthermore, increasing the distance between the light guide tip and RMC specimens did not effectively compensate for the light beam non-uniformity of LCUs. Additionally, for all LCUs, increased light beam non-uniformity significantly reduced the surface hardness of RMC specimens.

In 2008 Vandewalle et al.[18] assessed the effect of the light guide type on the distribution of irradiant emission from an LED LCU and evaluated the effect of the light dispersion on surface hardness of two types of RMC. In this study, the authors found a more uniform light distribution when using the standard guide than the turbo guide. Also, higher hardness values were found at higher irradiance locations on both top and bottom surfaces.

More recently, Price et al.[17] correlated the effects of irradiance and spectral emission inhomogeneities of a multiple emission peak LED LCU on the microhardness of RMC containing either camphorquinone (CQ) or a combination of CQ and trimethylbenzoyl-diphenylphosphine oxide (TPO) photoinitiator systems. In general, the irradiance and spectral emission were non-uniform across the LCU tip. However, the authors found a significant positive correlation between the irradiance beam profile and the microhardness values for RMC specimens cured for 5 seconds and 10 seconds. This correlation increased with RMC specimens containing both CQ and TPO photoinitiators and decreased with increasing curing time for RMC specimens containing CQ only. Haenel et al.[23] also investigated the effect of irradiance distribution of multiple emission peak and single emission peak LED LCUs on the microhardness of RMC. RMC hardness values correlated significantly with the irradiance distribution across the LCU tip.

Total radiant exposure is the product of irradiance and exposure time. Comparable material properties may result from similar radiant exposure regardless of how it was obtained (amount of irradiance, exposure time). This principle is known as the "exposure reciprocity law."[47] Price et al.[11] suggested that increasing the light curing time beyond the manufacturer recommendations might partially overcome the effect of light beam non-uniformity on the polymerization and microhardness of RMC.[11] However, Haenel et al.[23] found that extending the exposure time did not compensate for the effect of the light beam non-uniformity on the microhardness of RMC specimens, which confirms the findings of previous reports on exposure reciprocity.[47-49]

DEGREE OF CONVERSION AND FLEXURAL STRENGTH

Adequate polymerization of RMC is an essential requirement for long and predictable clinical performance.[38,50-52] Previous studies have evaluated the DC and flexural strength of different restorative materials to predict their clinical performance when subjected to mastication stresses after being introduced to the oral environment. Ferracane et al.[53] in 1985 found a significant positive correlation between increasing DC and microhardness values of RMC specimens.[53] Likewise, in 2003 Palin et al.[31] evaluated the DC and flexural strength of several RMC materials and found that lower DC values could significantly compromise the flexural strength properties of RMC.

RMC materials will attain their maximum physical and mechanical properties only if they receive adequate energy at an appropriate wavelength.[20] In 2006 Calheiros et al.[32] reported that increasing the radiant exposure could positively affect the flexural strength but not necessarily the DC of RMC. However, the authors emphasized the consequences of increasing the photoactivation of RMC, as it may result in temperature rise of the surrounding tissues.[32] Gonzalez et al.[54] investigated the effect of the irradiance and radiant exposure on the DC and the mechanical properties of two different RMC materials. Higher irradiance values significantly improved the flexural strength properties but did not influence the DC of RMC. Therefore, from the previous studies, it can be concluded that the flexural properties were found to be improving with adequate polymerization of RMC.[31,32, 54,55] However, the DC and flexural strength of RMC were also found to be significantly influenced by the material composition, as increasing the monomer concentration, viscosity and opaque filler loading could significantly decrease the DC of RMC. Also, the amount of the filler content of RMC could be influenced by

the filler morphology and may affect the flexural strength of RMC.[56-59]

ISOTROPIC AND ANISOTROPIC BEHAVIOR OF RMC

Polymers could have either completely non-crystalline (amorphous) or semi-crystalline structure.[60] However, several factors could affect the crystallinity of the polymer structure. Among those factors are the symmetry, intermolecular bonding, tacticity, molar mass, and branching of the polymer chain.[60] Linear chains with symmetrical units increase the ability of a polymer to crystalize.[60] Also, polymer chains that have large pendant groups will increase the rigidity and result in the difficulty to form a crystalline array.[60] Additionally, if the chain is significantly branched, the packing efficiency deteriorates and the crystalline content is lowered.[60] The crystallographic direction of a crystalline material is defined as a line between two atoms or a vector in the material crystal structure. The physical properties of some crystalline materials may depend on their crystallographic direction. For example the elastic modulus of a material may vary according to the atomic direction in its crystal structure, which means that the material may behave differently when subjected to forces in different directions.[61] The directionality of the properties is termed anisotropy.[61] However, materials with properties independent of their atomic direction are termed isotropic.[61] RMC with particulate fillers such as restorative and veneering composite is considered to have isotropic behavior because of its randomly oriented fillers. Thus, its properties are the same in all directions.[61] On the other hand, resin-impregnated fiber-reinforced composites were introduced to improve the mechanical and esthetic properties of RMC restorations.[62-64] However, the mechanical properties of fiber-reinforced composite may vary significantly according to the orientation of the fibers, as unidirectional fiber orientation produces

anisotropic properties, whereas randomly oriented fibers gives isotropic properties.[65]

In 2003 Dyer et al.[66] evaluated the influence of fiber position and orientation on fracture load of fiber-reinforced RMC using a three-point loading test. Both the orientation and the position of the fibers significantly influenced the fracture load to initial and final failure, as unidirectional glass fibers located on the tension side showed significantly higher fracture load values compared with fibers with diagonal orientation on the compression side.[66] Therefore, it is necessary to consider the type, the orientation, and the position of the fillers used in RMC, as they may affect the fracture load of RMC.

MATERIALS AND METHODS

MATERIALS

A quartz-tungsten-halogen (QTH) LCU (Optilux 401, Kerr, Orange, CA) (O), a multiple emission peak LED LCU (VALO Cordless, Ultradent, South Jordon, UT) (V) and a single emission peak LED LCU (Demi Ultra, Kerr, Orange, CA) (DU) were investigated (Figure 1). The diameters of the light guides for O, V and DU were 11 mm, 10 mm and 8 mm, respectively. A nano-hybrid resin-matrix composite, Tetric EvoCeram, (Bleaching shade XL, Ivoclar Vivadent, Amherst, NY) with a total inorganic filler content of 60% to 61% (by volume) and CQ and TPO photoinitiators was used in this study (Table I).

A Managing Accurate Resin Curing System-Resin Calibrator (MARC-RC) system (Bluelight Analytics Inc., Halifax, Canada) was used to monitor the amount, type, and rate of energy delivered to the top and bottom surfaces of RMC specimens (Figure 2).[67] The MARC-RC contains a National Institute of Standards and Technology (NIST)-referenced miniature spectrometer (USB4000, Ocean Optics, Dunedin, FL) with a 3,648-element linear CCD array detector (TCD1304AP, Toshiba, Tokyo, Japan).[68] The sensor is a CC3 cosine corrector (4-mm diameter) designed to collect radiation higher than a field of view of 180°, eliminating optical interface problems associated with the light collection sampling geometry.[68]

SAMPLE PREPARATION

The specimens were prepared in a constant temperature room (21°C) with 380-nm to 520-nm filtered ambient light.

Forty-five specimens were prepared and assigned to nine groups (n = 5) according to their light curing locations (LCLs) (1, 2, 3) (Table II) using a custom-made dark polyvinyl chloride (PVC) mold. The mold was designed with a rectangular opening centered over the 4-mm MARC-RC bottom sensor and an external shape that fit into the well of the bottom sensor to prevent mold rotation and to standardize the location of the specimens relative to each LCU (Figure 3). To analyze the effect of the beam profile of the LCUs on μ-flexural strength, rectangular RMC specimens were made with dimensions (2-mm width × 1-mm thickness × 6-mm length) allowing them to be polymerized in one exposure. The mold was placed on a Mylar strip and on top of a microscope slide. Then, Tetric EvoCeram bleaching shade XL was packed into the rectangular opening of the mold with a hand instrument and covered with another Mylar strip and a microscope slide to compress the excess resin (Figure 4). The mold with Mylar strips only was placed on the bottom sensor of MARC-RC. The light beam profile of each LCU was previously quantified using a beam profiler system combined with mean power measurements calculated from the irradiance values collected from a MARC-RC system (Figure 5).[69] Each LCU was mounted on a mechanical arm perpendicular to and centered over the top sensor of MARC-RC, and the irradiance and radiant exposure for each LCU were collected at three different LCLs: 1) at the center of the end of the tip; 2) at 1.5 mm to the left of the center of the end of the tip; and 3) at 1.5 mm to the right of the center of the end of the tip (Figure 6). Then, the LCU was shifted

over the bottom sensor without changing the alignment of the LCU (Figure 7). Each LCU was mounted to be parallel to the RMC specimens and at 0-mm distance between the top of the specimens and the light guide tip. For each LCU, specimens were polymerized for 10 seconds according to manufacturer recommendations (Figure 8), and the amount of irradiance and radiant exposure transmitted through the specimen were estimated during the photoactivation of each specimen. To simulate a clinical situation, RMC specimens were photoactivated from the top surface only. After that, the specimens were removed from the mold and placed in a well-plate containing deionized (DI) water and covered with aluminum foil to prevent light from reaching the specimen (Figure 9).

FINISHING AND POLISHING

Immediately after fabrication, specimens were mounted over a circular block and stabilized with molten wax applied around each specimen and the block was placed on the finishing and polishing machine. Specimens were finished using a Struers RotoForce-4 polishing unit (Struers, Ballerup, Denmark) using 1,200-, 2400- and 4,000-grit SiC abrasive paper for 1 second, 8 seconds, and 19 seconds respectively and with 5 N force on each specimen. Then, specimens were polished using 1-μm diamond polishing suspension (Struers, Ballerup, Denmark) and ultrasonically cleaned for 20 min in DI water. Specimens were stored wet in DI water at 37^{o}C for 24 hours.

DEGREE OF CONVERSION (DC)

An Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) spectrometer (ATR-MIRacle, Pike Technologies, Madison, WI, and JASCO 4100 International Co., Tokyo, Japan) was used to measure the DC. The ATR had a 1.8-mm

diameter diamond crystal plate. Uncured RMC specimens were measured (n = 3). The area under the 1608 cm^{-1} peak was assigned to the aromatic C=C, and the area under the 1637.5 cm^{-1} peak was assigned to the vinyl C=C.[35,37, 38,56] Cured RMC specimens were placed over the crystal and secured with a swivel pressure clamp to ensure adaptation. The absorbance was measured using 64 scans and 4 cm^{-1} resolution. Three measurements were obtained from different locations (A, B, C) on the top and bottom surface of each specimen (Figure 10). The average of the three measurements on the top surface was compared with the average of the three measurements on the bottom. The DC was calculated using the following equation[70]:

$$\text{Degree of conversion} = (1 - \frac{\text{cured (area under 1637.5/area under 1608)}}{\text{uncured (area under 1637.5/area under 1608)}}) \times 100$$

MICRO-FLEXURAL STRENGTH (μ-flexural strength)

Twenty-four hours after specimen preparation, a micro-three-point bending test using a universal mechanical testing machine (MTS, Eden Prairie, MN) at a crosshead speed of 1 mm/min with support span of 4 mm was used to determine μ-flexural strength (Figure 11). Flexural strength (σ) was calculated in megapascals (MPa) using the following equation:

$$\sigma = \frac{3Fl}{2bh^2}$$

Where *(F)* was the maximum load exerted on the specimens in Newtons; *(l)* was the distance between the supports in mm; *(b)* was the width of the specimen in mm,; *(h)* was the height of the specimen in mm.

STATISTICAL ANALYSIS

Irradiance, radiant exposure, and DC were analyzed using three-way ANOVA, with each LCU-LCL-surface combination allowed to have a different variance, and random effects to correlate multiple measurements from each specimen. The μ-flexural strength was analyzed using two-way ANOVA, with each LCU-LCL combination allowed to have a different variance. A 5-percent significance level was used for all tests.

RESULTS

IRRADIANCE AND RADIANT EXPOSURE

Tables III and IV, Figure 12 and Figure 13 demonstrate mean ±SE of the irradiance and radiant exposure for the evaluated LCUs at different LCLs. The bottom surface of a RMC specimen received significantly lower irradiance and radiant exposure compared with the top, regardless of the LCL ($p < 0.0001$). Comparing the irradiance received on a RMC specimen, LCL 1 received significantly higher irradiance than LCL 2 for top using DU ($p = 0.0033$) and V, regardless of the surface ($p = 0.0008$). LCL 1 received significantly higher irradiance than LCL 3 for top using DU ($p = 0.0045$) and V ($p = 0.0081$). LCL 2 received significantly higher irradiance than LCL 3 using DU regardless of the surface ($p = 0.0382$) and bottom using O ($p = 0.0153$). However, LCL 2 received significantly lower irradiance than LCL 3 for bottom using V ($p = 0.0024$). Comparing the LCUs, DU delivered significantly higher irradiance than O ($p < 0.0001$) and V ($p < 0.0001$). Also, V delivered significantly higher irradiance than O ($p < 0.0001$).

Comparing the radiant exposure, LCL 1 received significantly higher radiant exposure than LCL 2 for top using DU ($p = 0.0001$) and V, regardless of the surface ($p = 0.0009$), and significantly lower radiant exposure than LCL 2 for bottom using O ($p = 0.0333$). LCL 1 received significantly higher radiant exposure than LCL 3 for top using DU ($p = 0.0008$) and V ($p = 0.0176$). LCL 2 received significantly higher radiant exposure than LCL 3 for bottom using O ($p = 0.00383$). However, lower radiant exposure was received at LCL 2 than LCL 3 for top using DU ($p = 0.0175$) and bottom using V (p

= 0.0004). Comparing the LCUs, DU delivered significantly higher radiant exposure than O ($p < 0.0001$) and V ($p < 0.0001$), and V was significantly higher than O ($p < 0.0001$).

DEGREE OF CONVERSION (DC)

Table V and Figure 14 demonstrate the mean ±SE of DC for the evaluated LCUs at different LCLs. The bottom had significantly higher DC than the top for DU in LCL 1 ($p = 0.0113$). However, the bottom was significantly lower than the top at LCL 2 ($p = 0.0042$). Also, the bottom was significantly lower than the top for O regardless of LCL ($p = 0.0005$). And the bottom was significantly lower than the top for V at LCLs 2 and 3 ($p = 0.0001$). LCL 1 was significantly lower than 2 and 3 for the top of DU and V ($p < 0.0020$). The bottom of DU was significantly higher than O in all LCLs ($p = 0.0039$). Furthermore, DU was significantly higher than O on the top at LCL 2 ($p = 0.0017$). However, DU was significantly lower for the top at LCL 1 than O ($p = 0.0036$). DU was significantly higher than V for the bottom in all LCLs ($p = 0.0001$). O was significantly higher than V for top at LCL 1 ($p = 0.0004$) but was significantly lower at LCL 2 ($p = 0.0258$).

MICRO-FLEXURAL STRENGTH (μ-flexural strength)

Table VI and Figure 15 demonstrate the mean ±SE for μ-flexural strength (MPa) for the evaluated LCUs at different LCLs. DU was significantly lower than O ($p < 0.0450$) and V ($p < 0.0250$) for LCLs 1 and 3. However, LCL 1 was significantly lower than 2 for DU ($p = 0.0476$).

In general, locations that received higher irradiance exhibited higher radiant exposure values, but did not necessarily show higher DC or flexural strength values. In

addition, the DC values did not seem to influence the flexural strength of RMC specimens.

TABLES AND FIGURES

TABLE I

Composition of Tetric EvoCeram resin-matrix composite

Composition	**Weight (%)**
Bis-GMA, Urethane dimethacrylate, Ethoxylated Bis-EMA	16.8
Barium glass filler, Ytterbium trifluoride, Mixed oxide	48.5
Prepolymers	34.0
Additives	0.4
Catalysts and Stabilizers	0.3
Pigments	<0.1
Camphorquinone (CQ), trimethylbenzoyl diphenylphosphine oxide (TPO) photoinitiators	

TABLE II

Treatment groups and curing locations.

Group (Location)	**Light curing unit**	**Light curing tip location (mm)**	**N**
1	Multiple emission peak LED VALO Cordless (Test)	Center	5
2		1.5 mm to the left	5
3		1.5 mm to the right	5
1	Single emission peak LED Demi Ultra (Test)	Center	5
2		1.5 mm to the left	5
3		1.5 mm to the right	5
1	QTH Optilux-401 (Control)	Center	5
2		1.5 mm to the left	5
3		1.5 mm to the right	5

TABLE III

Mean ±SE of the irradiance (mW/cm^2) received by the resin-matrix composite using the evaluated LCUs at different locations*

		Mean (SE) irradiance (mW/cm^2)		
		Light Curing Location		
LCU	**Surface**	**1**	**2**	**3**
Single emission peak LED				
Demi Ultra$^{\alpha}$	Top^{a}	1336.9 (3.3)^{A}	1301.7 (1.4)^{B}	1296.8 (0.8)^{C}
	Bottom^{b}	86.5 (2.0)^{AB}	89.1 (2.2)^{A}	86.7 (1.1)^{B}
QTH				
Optilux$^{\gamma}$	Top^{a}	800.4 (9.8)^{A}	850.6 (16.7)^{A}	837.9 (13.0)^{A}
	Bottom^{b}	42.9 (0.4)^{AB}	45.8 (1.0)^{A}	41.5 (1.0)^{B}
Multiple emission peak LED				
Valo$^{\beta}$	Top^{a}	1314.0 (2.2)^{A}	1279.7 (4.1)^{B}	1276.7 (4.9)^{B}
	Bottom^{b}	62.1 (1.4)^{A}	53.4 (1.5)^{B}	63.0 (0.4)^{A}

*Superscript lowercase letters represent statistical groups between top and bottom surfaces. Superscript uppercase letters represent statistical groups in the same row. Superscript Greek letters represent statistical groups between LCUs. Abbreviations: Location 1: The center of the end of the tip. Location 2: LCU moved 1.5 mm to the left of the center of the end of the tip. Location 3: LCU moved 1.5 mm to the right of the center of the end of the tip.

TABLE IV

Mean ±SE of the radiant exposure (J/cm^2) received by the resin-matrix composite using the evaluated LCUs at different locations*

		Mean (SE) radiant exposure (J/cm^2)		
		Light Curing Location		
LCU	**Surface**	**1**	**2**	**3**
Single emission peak LED				
Demi Ultra$^{\alpha}$	Top^{a}	13.576 (0.025)^{A}	13.267 (0.002)^{C}	13.374 (0.032)^{B}
	Bottom^{b}	0.871 (0.021)^{A}	0.909 (0.023)^{A}	0.883 (0.012)^{A}
QTH				
Optilux-401$^{\beta}$	Top^{a}	8.206 (0.142)^{A}	8.728 (0.199)^{A}	8.633 (0.210)^{A}
	Bottom^{b}	0.417 (0.005)^{B}	0.448 (0.011)^{A}	0.402 (0.010)^{B}
Multiple emission peak LED				
VALO$^{\gamma}$	Top^{a}	13.337 (0.001)^{A}	13.246 (0.029)^{B}	13.240 (0.022)^{B}
	Bottom^{b}	0.609 (0.018)^{A}	0.541 (0.016)^{C}	0.638 (0.005)^{B}

Superscript lowercase letters represent statistical groups between top and bottom surfaces. Superscript uppercase letters represent statistical groups in the same row. Superscript Greek letters represent statistical groups between LCUs. Abbreviations: Location 1: The center of the end of the tip. Location 2: LCU moved 1.5 mm to the left of the center of the end of the tip. Location 3: LCU moved 1.5 mm to the right of the center of the end of the tip.

TABLE V

Mean ±SE of degree of conversion for the evaluated LCUs at different locations.

		Mean (SE) degree of conversion		
		Light Curing Location		
LCU	**Surface**	**1**	**2**	**3**
Single emission peak LED				
Demi Ultra	Top	54.3 (3.0)$^{bB\beta}$	65.8 (2.0)$^{aA\alpha}$	62.3 (1.1)$^{aA\alpha}$
	Bottom	60.7 (2.7)$^{aA\alpha}$	57.5 (1.9)$^{bA\alpha}$	58.5 (2.2)$^{aA\alpha}$
QTH				
Optilux-401	Top	61.5 (0.8)$^{aA\alpha}$	57.6 (0.4)$^{aA\beta}$	57.8 (2.3)$^{aA\alpha}$
	Bottom	55.2 (1.2)$^{bA\beta}$	52.9 (1.9)$^{bA\beta}$	52.3 (2.6)$^{bA\beta}$
Multiple emission peak LED				
VALO	Top	52.0 (0.6)$^{aB\beta}$	63.0 (1.3)$^{aA\alpha}$	60.6 (1.8)$^{aA\alpha}$
	Bottom	52.4 (2.4)$^{aA\beta}$	50.5 (2.4)$^{bA\beta}$	49.7 (1.5)$^{bA\beta}$

*Superscript lowercase letters represent statistical groups between top and bottom surfaces for each LCU. Superscript uppercase letters represent statistical groups in the same row. Superscript Greek letters represent statistical groups between LCUs for each surface. Abbreviations: Location 1: The center of the end of the tip. Location 2: LCU moved 1.5 mm to the left of the center of the end of the tip. Location 3: LCU moved 1.5 mm to the right of the center of the end of the tip.

TABLE VI

Mean ±SE for μ-flexural strength (MPa) for the evaluated LCUs at different locations*

	Mean (SE) μ-flexural strength (MPa) Light Curing Location		
LCU	**1**	**2**	**3**
Single emission peak LED			
Demi Ultra	313 (31)^{bB}	400 (13)^{aA}	369 (10)^{bAB}
QTH			
Optilux-401	401 (12)^{aA}	425 (16)^{aA}	405 (2)^{aA}
Multiple emission peak LED			
VALO	417 (13)^{aA}	418 (5)^{aA}	458 (21)^{aA}

*Superscript lowercase letters represent statistical groups in the same column. Superscript uppercase letters represent statistical groups in the same row. Abbreviations: 1: The center of the end of the tip. Location 2: LCU moved 1.5 mm to the left of the center of the end of the tip. Location 3: LCU moved 1.5 mm to the right of the center of the end of the tip.

FIGURE 1. Top left: A single emission peak LED LCU (Demi Ultra). Top right: A multiple emission peak LED LCU (VALO Cordless). Bottom: A QTH LCU (Optilux-401).

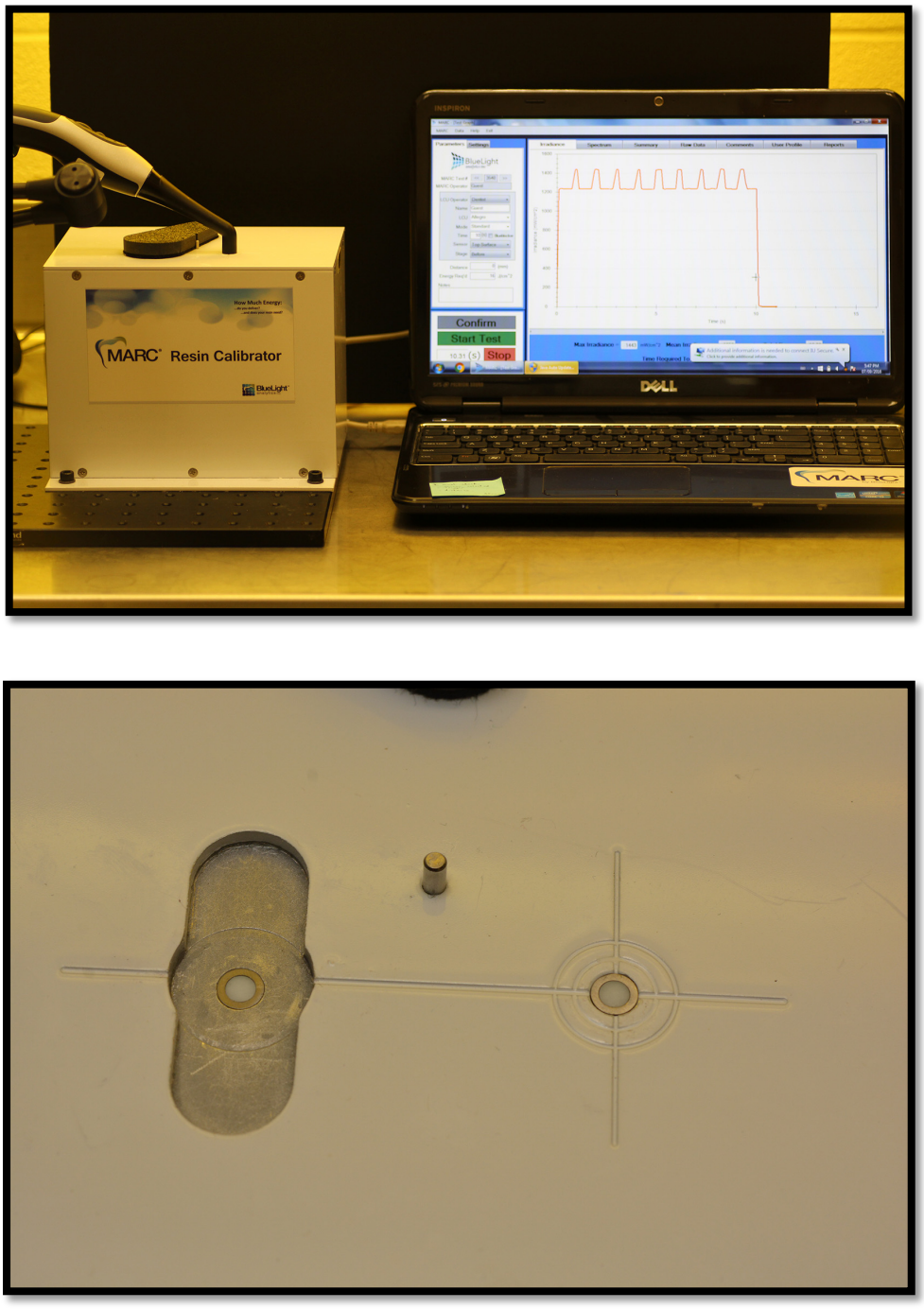

FIGURE 2. Top: A Managing Accurate Resin Curing System-Resin Calibrator (MARC-RC) system. Bottom: MARC-RC top sensor (right) and bottom sensor (left).

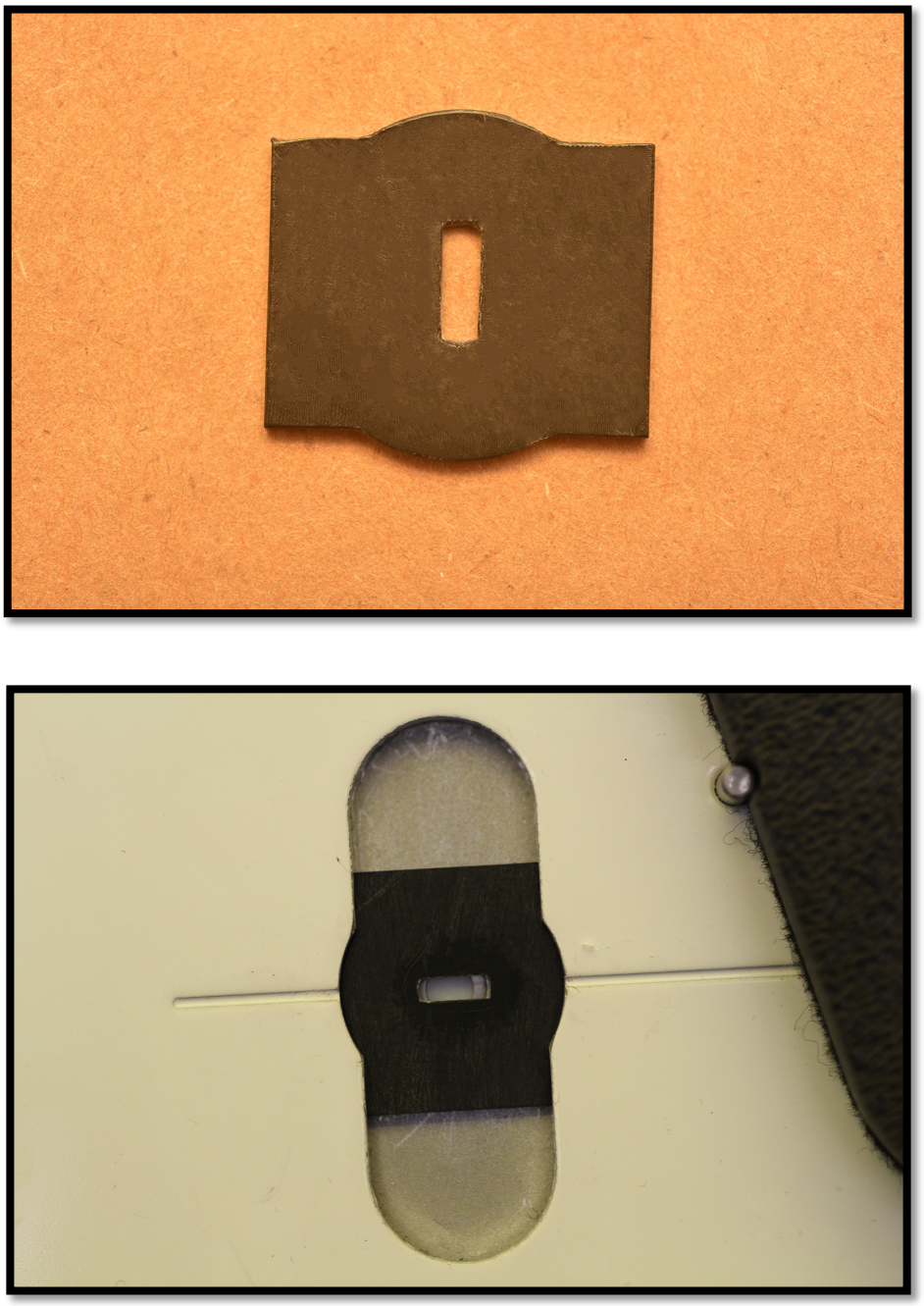

FIGURE 3. Top: Polyvinyl chloride (PVC) dark mold that has a rectangular opening with dimensions of (2mm width × 1 mm thickness × 6mm length) to fabricate RMC specimens. Bottom: The rectangular opening of the PVC mold centered over the 4 mm MARC-RC bottom sensor.

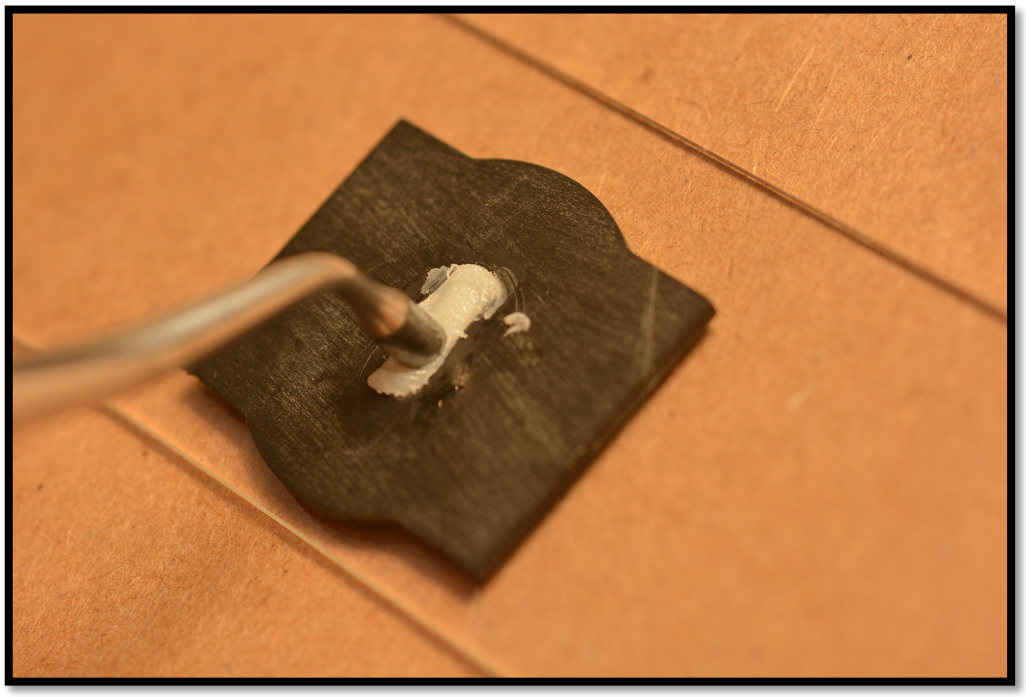

FIGURE 4. Illustration of the RMC packed into the rectangular opening of the mold with a hand instrument.

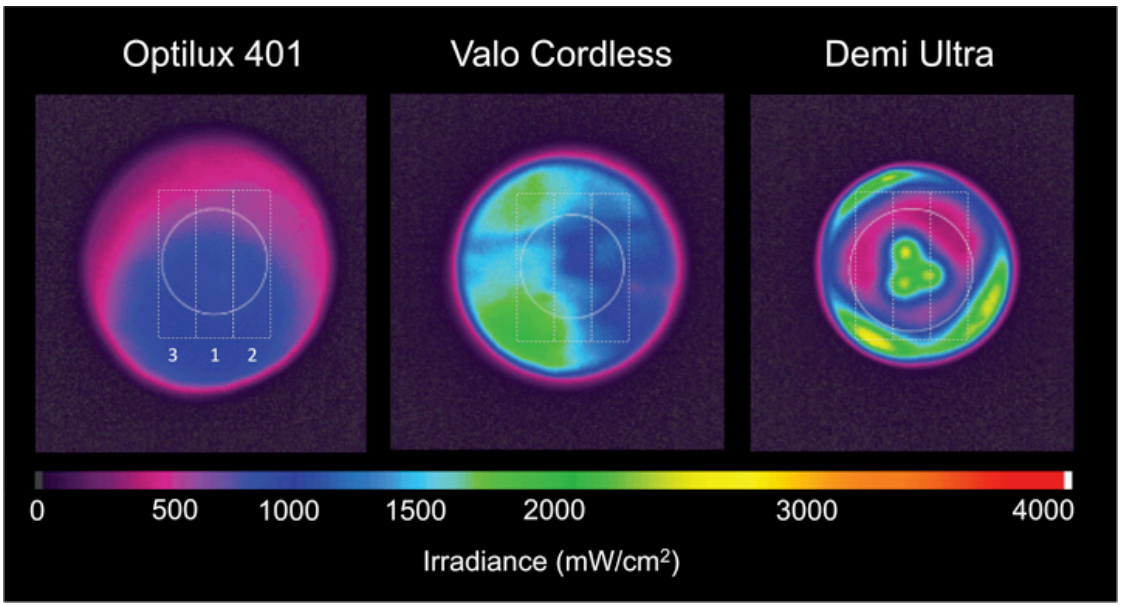

FIGURE 5. Illustration of the specimen locations where the measurements were collected relative to each light-curing unit (LCU) tip. The circle in the center of each image represents the 4-mm MARC-RC sensor where the irradiance measurements were collected. The dashed lines represent the light-curing unit locations where the light was irradiated and different measurements were collected. Numbers represent the specimen location. Abbreviations: Location 1: The center of the end of the tip. Location 2: LCU moved 1.5 mm to the left of the center of the end of the tip. Location 3: LCU moved 1.5 mm to the right of the center of the end of the tip.

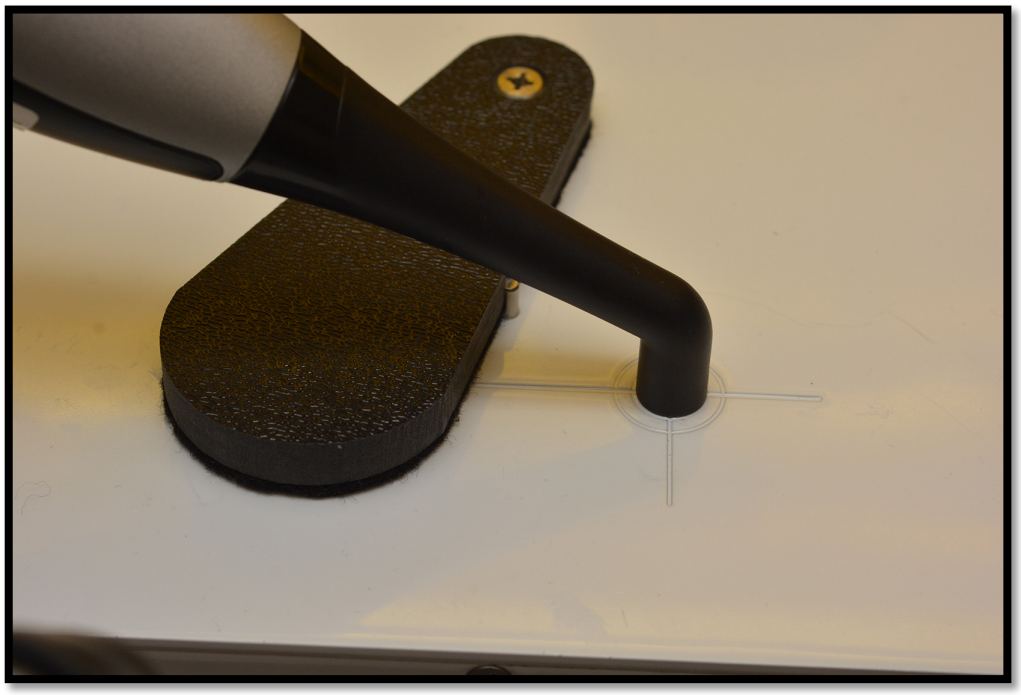

FIGURE 6. Illustration of the LED LCU mounted over the top sensor of MARC-RC to measure the amount of irradiance and radiant exposure received by the top surface of RMC specimen.

FIGURE 7. Illustration of the LED LCU mounted over the bottom sensor of MARC-RC and at 0-mm distance between the top of the specimen and the light guide to measure the amount of irradiance and radiant exposure received by the bottom surface of RMC specimen.

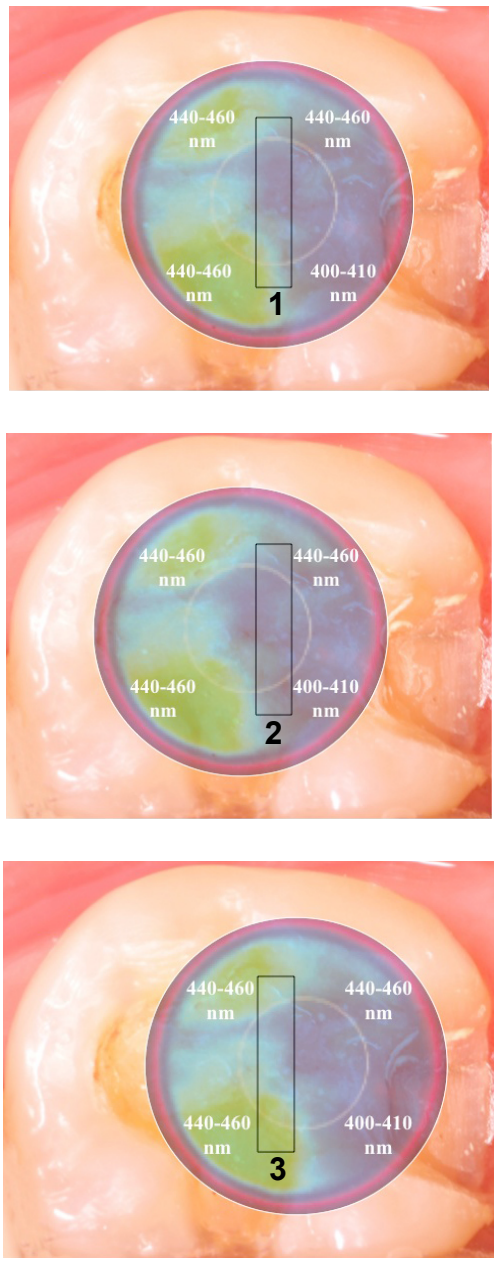

FIGURE 8. Illustration of the specimen locations relative to the beam profile of the LCU and superimposed over a Class II cavity preparation at different LCLs (1, 2, 3). Three irradiance hotspots deliver higher irradiance at spectral emission of 440 460 nm and one hotspot delivers lower irradiance at spectral emission of 400-410 nm. 1: The center of the end of the tip. Location 2: LCU moved 1.5 mm to the left of the center of the end of the tip. Location 3: LCU moved 1.5 mm to the right of the center of the end of the tip.

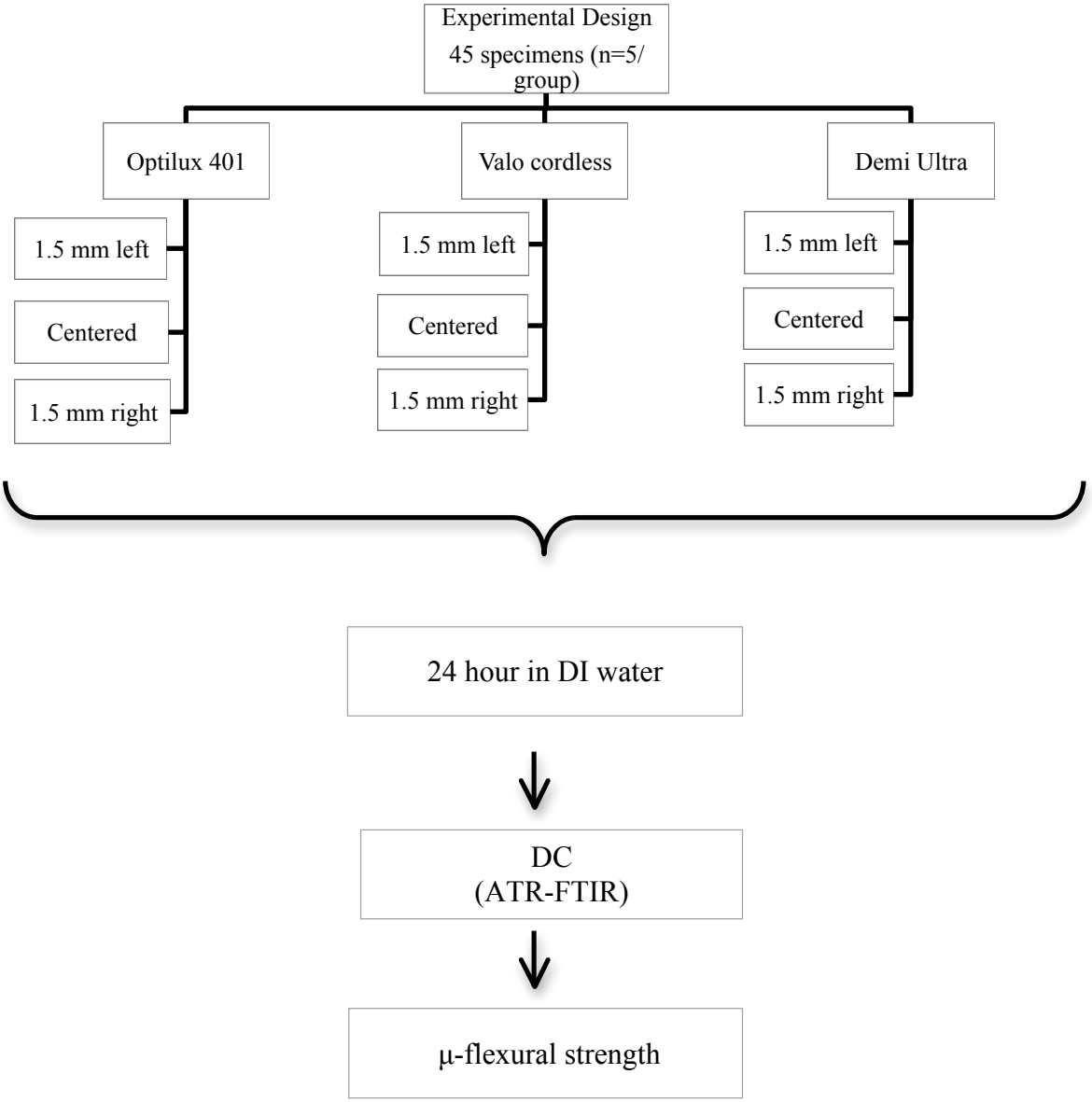

FIGURE 9. Flowchart of the experimental design. Abbreviations. DC: Degree of conversion. μ-flexural strength: micro-flexural strength.

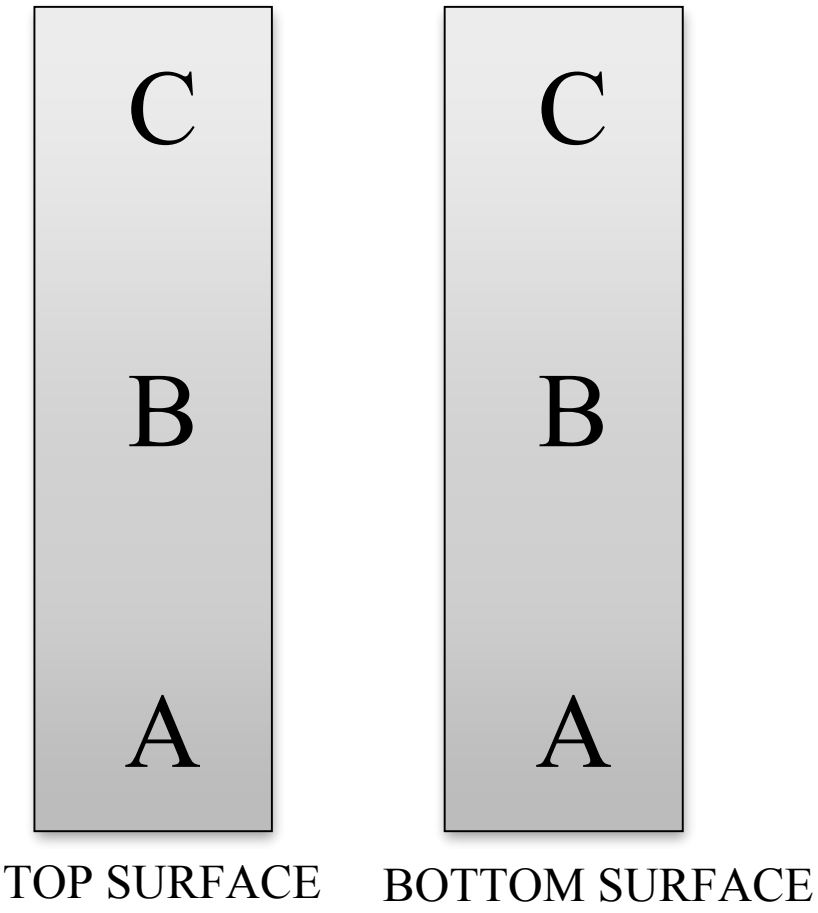

FIGURE 10. Illustration of the degree of conversion (DC) measurement locations on the top and bottom surfaces of a RMC specimen. A: DC measurement below the center of a RMC specimen. B: DC measurement at the center of a RMC specimen. C: DC measurement above the center of a RMC specimen.

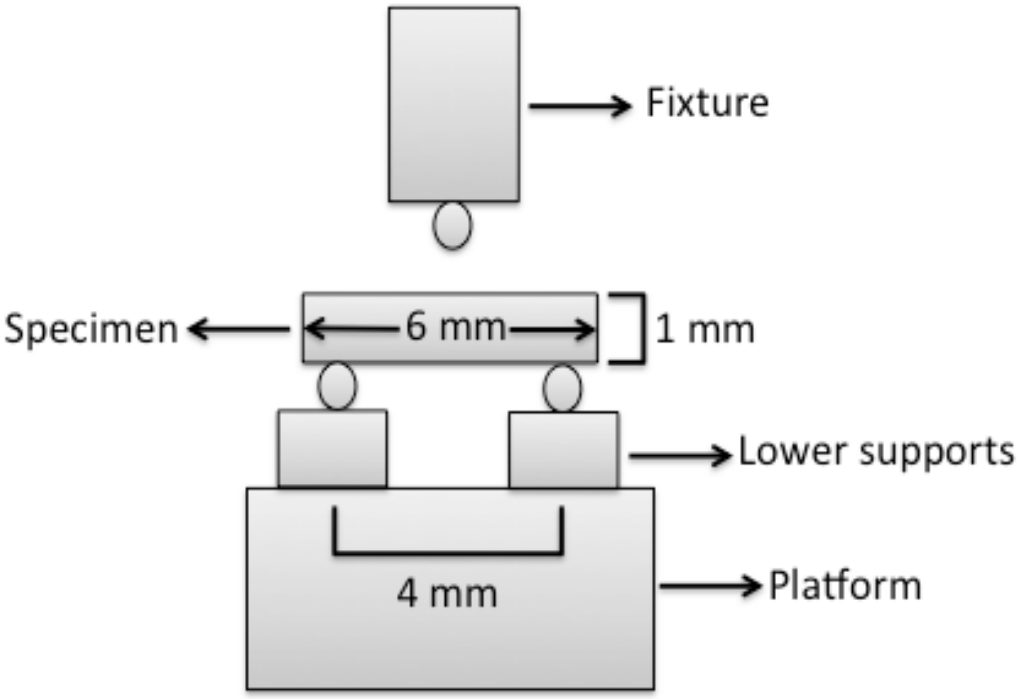

FIGURE 11. Illustration of μ-flexural strength test setup.

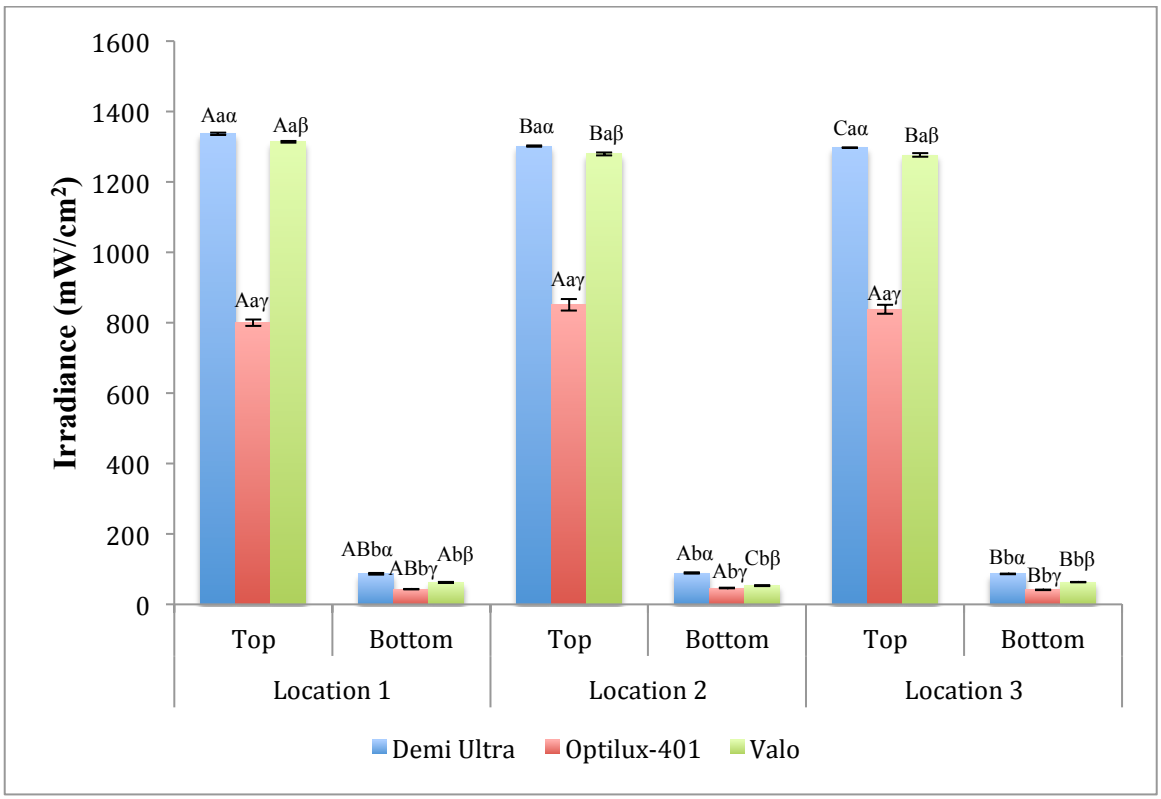

FIGURE 12. Mean ±SE of the irradiance (mW/cm^2) for the evaluated LCUs at different locations. Location 1: The center of the end of the tip. Location 2: LCU moved 1.5 mm to the left of the center of the end of the tip. Location 3: LCU moved 1.5 mm to the right of the center of the end of the tip. Uppercase letters represent statistical groups between locations for each LCU. Lowercase letters represent statistical groups between top and bottom surfaces. Greek letters represent statistical groups between LCUs.

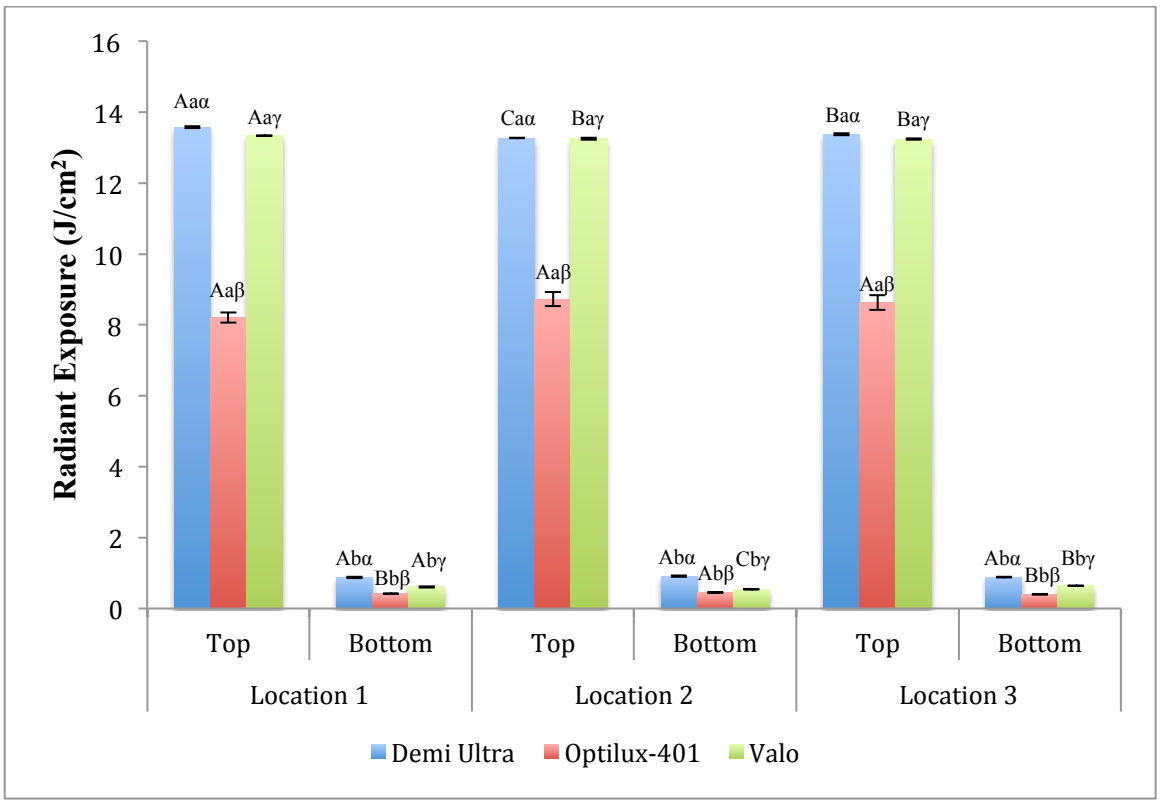

FIGURE 13. Mean ±SE of the radiant exposure (J/cm^2) for the evaluated LCUs at different locations. Location 1: The center of the end of the tip. Location 2: LCU moved 1.5 mm to the left of the center of the end of the tip. Location 3: LCU moved 1.5 mm to the right of the center of the end of the tip. Uppercase letters represent statistical groups between locations for each LCU. Lowercase letters represent statistical groups between top and bottom surfaces. Greek letters represent statistical groups between LCUs.

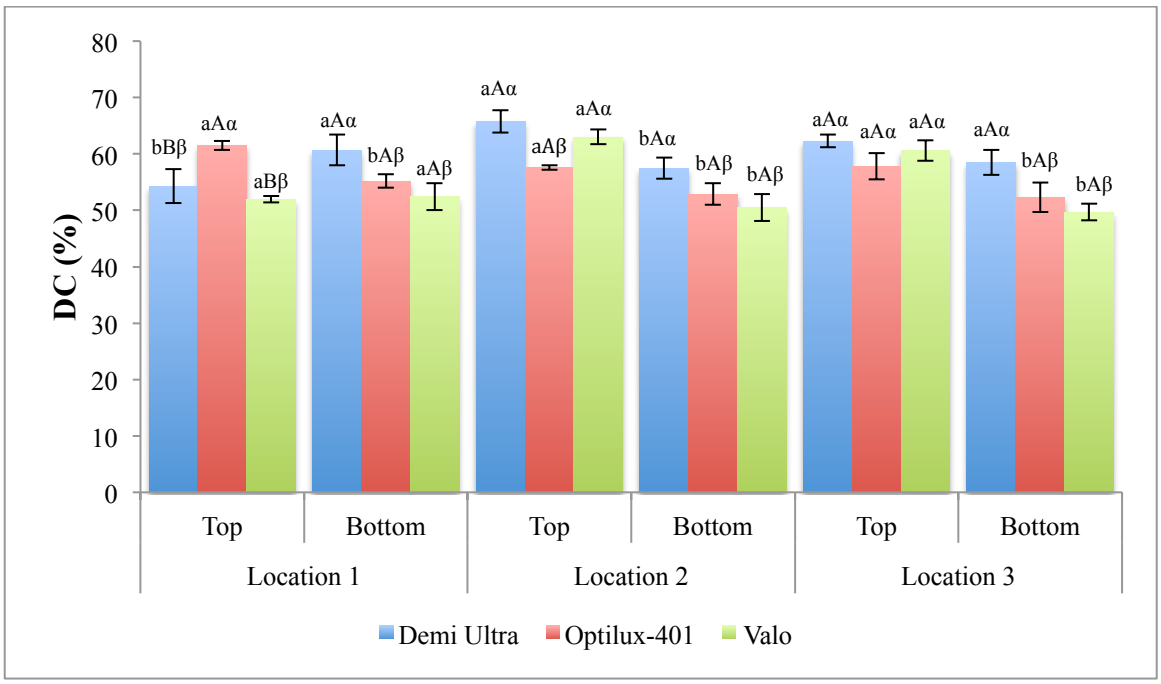

FIGURE 14. Mean ±SE of DC for the evaluated LCUs at different locations. Location 1: The center of the end of the tip. Location 2: LCU moved 1.5 mm to the left of the center of the end of the tip. Location 3: LCU moved 1.5 mm to the right of the center of the end of the tip. Lowercase letters represent statistical groups between top and bottom surfaces for each LCU. Uppercase letters represent statistical groups between locations for each LCU. Greek letters represent statistical groups between LCUs for each surface.

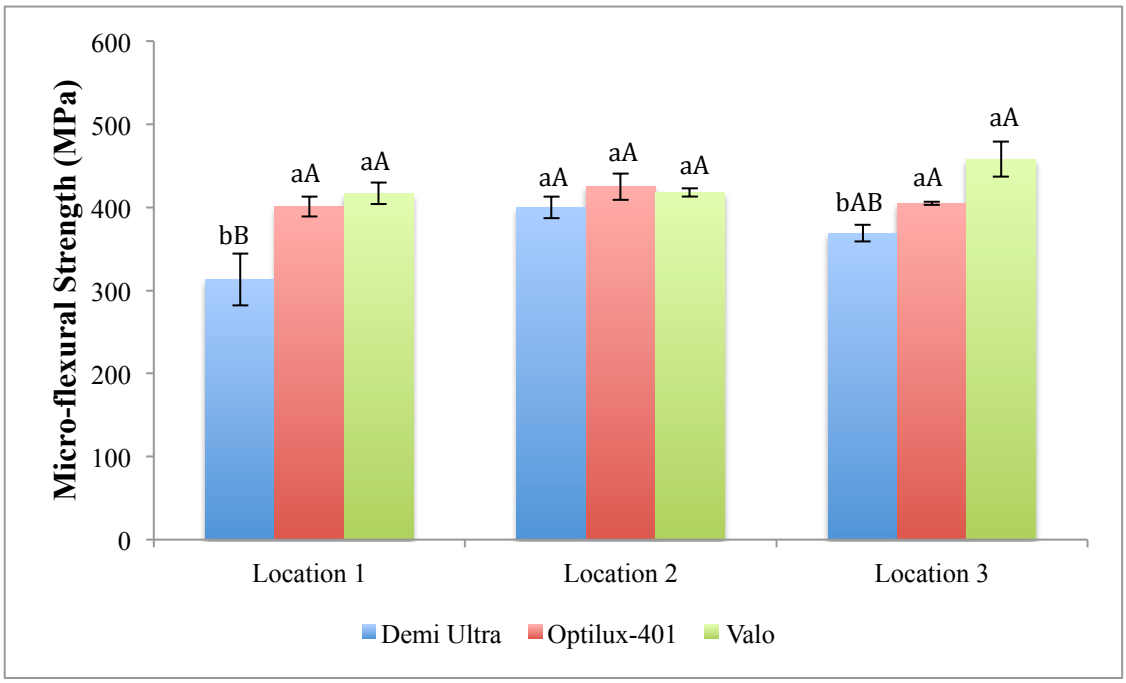

FIGURE 15. Mean ±SE of μ-flexural strength (MPa) for the evaluated LCUs at different locations. Location 1: The center of the end of the tip. Location 2: LCU moved 1.5 mm to the left of the center of the end of the tip. Location 3: LCU moved 1.5 mm to the right of the center of the end of the tip. Lowercase letters represent statistical groups between LCUs within the same location. Uppercase letters represent statistical groups between locations for each LCU.

DISCUSSION

IRRADIANCE AND RADIANT EXPOSURE

The ISO 4049 Standard for flexural strength[71] test suggests specimen dimensions of 2-mm width × 2-mm thickness × 25-mm length. However, specimens of this size require multiple overlapping irradiation cycles. Therefore, in this study, to assess the effect of non-uniform polymerization of RMC, a micro-flexural strength test was used. However there is no ISO standard for specimen dimensions of the micro-flexural strength test.[33,35,37,72,73] Also, the 16:1 proportion of the span to depth ratio that was suggested to minimize shear and local deformation effects was impractical and could not be used in this study, as the specimens became very thin and more prone to fracture during preparation.[35] Therefore, the dimensions were modified to 2-mm width × 1-mm thickness × 6-mm length to allow the light guide to completely cover the sample length, thereby curing samples in a single exposure.

For each LCU, it is important to note that the irradiance was collected from the 4-mm MARC-RC cosine corrector, which does not represent the effective light-emitting portion of the LCU tip. The significant differences observed between the LCL may be attributed to the differences in the beam profile radiated from each LCU. Also, RMC specimens cured at 0-mm distance from the LCU tip may not represent an actual clinical scenario and may receive higher irradiance and radiant exposure on the top and bottom surfaces.

Figure 2 shows higher irradiance at the center of the LCU tip for both DU and V. DU decreased on either side of the LCU, while V decreased only on the right side of the center. That could explain the significantly higher irradiance and radiant exposure delivered to the top surface at LCL 1 compared with LCLs 2 and 3 using DU and bottom at LCL 1 compared with LCL 2 using V. In addition, it could also explain the significantly lower irradiance and radiant exposure observed on the bottom at LCL 2 compared with LCL 3 using V. However, the presence of the LED violet chip in the lower right side of the multiple emission peak LED LCU tip may explain the lower irradiance and radiant exposure values on bottom surfaces at LCL 2. Although the irradiance is higher to the left of the center of the light curing unit tip in V, the significantly higher irradiance and radiant exposure at LCL 1 compared with LCL 3 on the top could be related to the convexity of the tip, which may focus the irradiance at the center of the light guide tip. Also, the higher irradiance delivered at LCL 2 compared with LCL 3 on the top and bottom using DU could be attributed to the larger area of high irradiance that covered the specimens at LCL 2. Although significantly lower radiant exposure was delivered on the top using DU at LCL 2 and 3, these differences may not be clinically significant. Even though O had relatively the most uniform light beam profile, the slight differences in the irradiance across the light beam profile could explain the significant differences observed in irradiance and radiant exposure on the bottom surfaces of RMC specimens.

DEGREE OF CONVERSION (DC)

Polymerization of RMC is mainly dependent on the curing conditions and material composition, which may affect their mechanical properties.[74-77] One of the

factors that affect the polymerization of a RMC is the photoinitiator system. CQ is the most common photoinitiator used in RMC.[78] Alternative photoinitiators, such as TPO, were introduced to overcome the yellow color of CQ.[26,79,80] As a result, multiple emission peak LED LCUs, which include an additional peak within the absorption range of TPO (350 nm to 425 nm) were developed.[81] Multiple emission peak blue-violet LED LCUs contain blue and violet chips, with spectral emission peaks of 440 nm to 460 nm and 400 nm to 410 nm, respectively, which have been suggested to improve the polymerization of TPO-containing RMC.[26,82,83] The RMC used in this study contains the TPO photoinitiator system, which may have different polymerization behavior than CQ and could result in higher polymerization rates compared with non-TPO RMC.[84] However, the bleaching shade used in the present study may improve the light transmission through the specimens and consequently result in higher DC values compared with darker shades.[85,86] On the other hand, the thickness of the specimens (1 mm) used in the present study may allow more light transmission through the material and result in higher DC values on bottom surfaces of specimens compared with the maximum thickness (2 mm) suggested for each RMC increment.[87,88]

Pigments and photoinitiators may decrease the amount of the light transmitted through RMC restorations; this may explain the same or significantly lower DC on the bottom of a specimen compared with the top. [85, 86] However, the significantly higher DC on the bottom compared with the top using DU at LCL 1 may be explained by the light reflected off the radiopaque MARC-RC bottom sensor to the bottom surfaces.

The lower DC values with DU on the T at LCL 1 compared to LCL 2 and 3 could be related to the high amount of free radicals produced from RMC specimens, which may

result in higher rate of polymerization that rapidly increased the viscosity of the dimethacrylate monomer system and consequently restricted the mobility of the molecules, resulting in more free radical entrapment, increased termination, and lower DC values.[38] In addition, the lower DC values with V on the top at LCL 1 compared with LCL 2 and 3 could be related to the presence of the LED violet chip at LCL 2.[19, 26-28]

The significantly higher DC values of DU compared to O and V on the bottom at all LCLs can be attributed to the higher irradiance and radiant exposure delivered with DU. This result confirms previous findings on the correlation of irradiance and radiant exposure with DC.[32, 54] Also, the higher DC on the top using O compared with DU and V at LCL 1 could be related to the broad spectral emission and uniform light beam profile of O LCU. However, the higher DC on the top using DU and V compared with O at LCL 2 could be attributed to the high irradiance and radiant exposure delivered by DU and V compared with O.[32,54]

MICRO-FLEXURAL STRENGTH (μ-flexural strength)

The monomer concentration, viscosity, and inorganic filler morphology may also affect the DC and flexural strength of RMC, which vary among different types of RMC.[56-59] The significantly lower μ-flexural strength using DU at LCL 1 than 2 may be related to the rate of polymerization, as the TPO may not be efficiently activated because higher irradiance was delivered to the sample within the CQ range and not the TPO range (violet range) and could result in lower cross linked density, which could influence the mechanical properties of RMC.[19,26-28,38] These results may confirm the previous findings on the correlation between adequate polymerization and the μ-flexural strength of RMC.[31,32,54,55] The significantly lower μ-flexural strength for DU compared with O and V

at LCL 1 and 3 could be related to the broad spectral emission of O and V LCUs because a QTH LCU has a more uniform light beam profile than LED LCUs, which were reported to be non-uniform.[20,46]

Among the limitations of this study, only one type of RMC was analyzed. Therefore, due to the large diversity of RMC products, it might be difficult to generalize the results of this study to other RMCs. Instead, our results may provide a reference about the possible impact of light beam profile non-uniformity on the polymerization and mechanical properties of RMC materials.

The findings of this study demonstrated significant variations in the DC and flexural strength of a RMC. Therefore, more studies are needed to further investigate and understand this possible detrimental effect. Furthermore, besides the importance of optimizing the exposure time and distance between the LCU tip and RMC restoration, clinicians should consider the type of photoiniators used in RMC relative to the spectral emission of various types of LCUs present in the market.

Future studies should continue to evaluate the effect of light beam non-uniformity of different LED LCUs on the mechanical properties of RMC cured at several distances from LCU tip and with various exposure times and photoinitiator systems. Also, future work should examine the variation of DC by mapping DC values across the top and bottom surfaces of RMC specimens to provide an accurate representation on the effect of the light beam non-uniformity on the polymerization of RMC restorations.

SUMMARY AND CONCLUSIONS

This study showed that bottom surfaces of RMC specimens received significantly lower irradiance and radiant exposure than top surfaces irrespective of LCL or LCU. All LCUs exhibited significant differences in the DC values between top and bottom surfaces at all LCLs except for V LCL 1 and DU LCL 3. Also, DU and V showed lower DC values on top surfaces at LCL 1 than 2 and 3. The μ-flexural strength was significantly lower for DU LED LCU at LCL 1 than LCL 2. Therefore, the non-uniform light curing beam profile could have a significant effect on μ-flexural strength and DC of RMC specimens cured at different LCLs.

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ABSTRACT

INFLUENCE OF CURING-LIGHT BEAM PROFILE NON-UNIFORMITY ON DEGREE OF CONVERSION AND MICRO-FLEXURAL STRENGTH OF RESIN-MATRIX COMPOSITE

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Background. Beam profile non-uniformity of light-curing units (LCUs) may result in suboptimal properties of resin-matrix composite (RMC) restorations. Objectives: The objective of this study was to evaluate the effect of curing-light beam profile of multiple light curing units (LCUs) on the degree of conversion (DC) and micro-flexural strength (μ-flexural strength) of RMC. Methods: Forty-five nano-filled hybrid RMC (Tetric EvoCeram, Ivoclar Vivadent, Amherst, NY) specimens were fabricated. Quartz-tungsten-halogen (QTH) (Optilux 401) (O), multiple emission peak (VALO Cordless) (V) and single emission peak (Demi Ultra) (DU) light-emitting-diode (LED) LCUs were

investigated at different light-curing locations (LCLs): 1) the center of the LCU tip; 2) 1.5 mm to the left of the center of the LCU tip; and 3) 1.5 mm to the right of the center of the LCU tip. Specimens were stored wet in deionized water at 37^{o}C for 24 hours. The DC was measured on top and bottom surfaces using Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) spectroscopy. Micro-flexural strength testing was performed using a universal mechanical testing machine at crosshead speed of 1 mm/min. Multi-factorial ANOVAs were used to analyze the data ($\alpha = 0.05$). Results: All LCUs exhibited significant differences in DC between top and bottom surfaces at the different LCLs (Table I). Micro-flexural strength varied with LCL for DU (Table II). Conclusions: The non-uniform curing-light beam profile could have a significant effect on μ-flexural strength and DC on top and bottom surfaces of RMC specimens cured at different LCLs.
Key words. LED, QTH, Mechanical properties.

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